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Determination of Physiochemical Properties of Biosorbents Synthesized from Water Melon Rind Using Microwave Assisted Irradiation Procedure

Augustus Newton Ebelegi, Enudi Ishioma Toneth, Makbere Anthony Bokizibe

Department of Chemical Sciences, Niger Delta University, Wilberforce Island, Bayelsa, Nigeria
Email: ebelegi@gmail.com

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Abstract

Three biosorbents were prepared from Watermelon Rind (WMR) using microwave assisted procedure and then characterized using physiochemical techniques (pH, bulk density, volatile matter, surface area, ash content and moisture content). Physiochemical characterization of the synthesized biosorbents was done in order to evaluate their adsorption potentials. Accordingly, results obtained from the experiments conducted revealed the following trend: pH: Water Melon Rind treated with Sodium hydroxide (NaWMR) 8.5 > Water Melon Rind treated with Hydrogen peroxide (HP-WMR) 8.1 > Water Melon Rind treated with Distilled water (DWMR) 6.4 > Untreated Water Melon Rind (UWMR) 5.4, which suggest that NaWMR and HP-WMR possess suitable pH values for the uptake of cationic species within aqueous systems. Surface Area: analysis: UWMR (21.4 m^2/g), DWMR (35.8 m^2/g), NaWMR (40.6 m^2/g) and HP-WMR (61.4 m^2/g). This means that HP-WMR has a larger surface area and could be a preferred candidate for adsorption processes. The results obtained from this study suggest that chemical modification of Water Melon Rind (WMR) with either distilled water Sodium Hydroxide or Hydrogen peroxide by means of microwave irradiation enhances physiochemical properties which could boost the adsorption capacity of Water Melon Rind. Thus, the outcome shows that all the three synthesized biosorbents; DWMR, NaWMR and HP-WMR possess the characteristics of a good adsorbent. Accordingly, they can be applied to wastewater treatment process.

Keywords

Physicochemical, pH, Distilled Water, Sodium Hydroxide, Hydrogen Peroxide, Bulk Density, Moisture Content

1. Introduction

Agricultural wastes usually termed agrowastes are residues obtained from growing and processing raw agricultural products such as vegetables, meat, fruits, milk products, and crops. They can also be aptly described as the non-product outputs of production and processing of agricultural products that may contain materials that are beneficiary to man but whose economic values are deemed to be less than the cost of collection, transportation, and processing for man's use. The make-up of agrowastes depends mostly on the system and type of agricultural activities and they are usually found as liquids, slurries, or solids.

Agricultural waste basically consists of animal waste, food processing waste (for instance approximately 20% of maize is canned while 80% is waste), crop waste (corn stalks, sugarcane bagasse, drops and culls from fruits and vegetables). Increasing agricultural production has naturally occasioned increased quantities of livestock waste, agricultural crop residues and agro-industrial by-products. Thus, there is likely to be a substantial upsurge in the generation of agricultural wastes globally as countries continue to boost farming systems. Reports have shown that approximately 998 million tons of agricultural waste is produced globally every year [1]. It is also on record that organic wastes can amount up to 80 percent of the entire solid wastes generated in most farms [2] [3]. Ultimately the waste generated is mostly dependent on the type of agricultural activities carried out.

Effluent discharges from industries typically release heavy metals such as Ni^{2+}, Zn^{2+}, Cu^{2+}, Cr^{2+} and Pb^{2+} into the aquatic environment [4].

In advanced climes, heavy metals found in waste water are ordinarily removed by advanced technologies such as vacuum evaporation, ion exchange resins, crystallization, membrane technology and solvent extraction. Conversely, in developing countries, the aforementioned treatment procedures cannot be carried out because of deficiency in technology and insufficient funds [5]. Therefore, it is desired that simple and eco-friendly heavy metal sequestration procedures be established and used in developing countries. Agricultural waste materials are proven to be both user and environmentally friendly with appreciable biosorption capacity too. Some basic advantages of using agricultural wastes as adsorbents include regeneration and recycling of bio-sorbents, high efficiency and low cost [6] [7].

Agricultural wastes obtained from plants contain loose and porous structures with functional groups such as carboxyl and hydroxyl groups that are responsible for their adsorption capacity [8]. Bio-sorbents generated from agricultural waste possess a strong affinity and high selectivity towards heavy metals due to availability of functional groups (binding sites) on their surfaces. They can also be easily processed, applied and recovered without posing undesirable impacts on the environment. [9].

Watermelon (*citrullus lanatus*) is a flowering plant species of the Cucurbitaceae family, the gourd family of flowering plant, belongs to the order Cucurbitales which encompasses about 975 species of food and ornamental plants. Members of this family of crops are annual or perennial herbs which are native to temperate and tropical areas they include cucumbers, gourds, melons, squashes, and pumpkins family. Watermelon is about the largest and heaviest fruit and one of the most munificent and inexpensive available fruit. The large fruit is a kind of modified berry called a pepo, with a thick rind (exocarp) and fleshy Centre (mesocarp and endocarp).

Watermelon rind (WR) consists of pectin, citrulline, cellulose, proteins and carotenoids which are rich in functional groups such as hydroxyl (cellulose) and carboxylic (pectin) [10]. Watermelon biomass can be characterized into three main components which are the flesh, seed and rind (**Figure 1**). The flesh constitutes approximately 65% of the total weight, and the rind constitutes approximately 30% and the seed about 2% [11].

Waste management has been a serious problem, mostly in the developing world, because of the treatment cost [12]. Open dumping and burning has been a common disposal practice of solid waste in Nigeria, and it is detrimental to human health as well as the environment.

Agricultural waste such as peels of fruits and vegetables contribute a large quantity of solid agrowastes and the high demand of watermelon owing to its health benefits has instigated the disposal of high volume of watermelon rind (WMR) found in most environment so there is need to generate adsorbents from it. The aim of this research work is to synthesize and characterize a bio-sorbent from watermelon rind (WMR). This study focused on the synthesis of two absorbents from watermelon rind and determined the physiochemical parameters of the adsorbents.

Biosorption may be defined as the removal of substances (adsorbates) from solution by biological materials, and the term could also mean the passive binding of metal ions or radioactive elements by dead biomass. Studies have shown that microorganisms such as fungi, algae, bacteria, yeast and plants can remove some pollutants from aqueous solutions and such biological phenomena are called biosorption. [13].

The uptake of both metal and non-metal species by biomass, whether living or denatured is commonly termed biosorption [14].

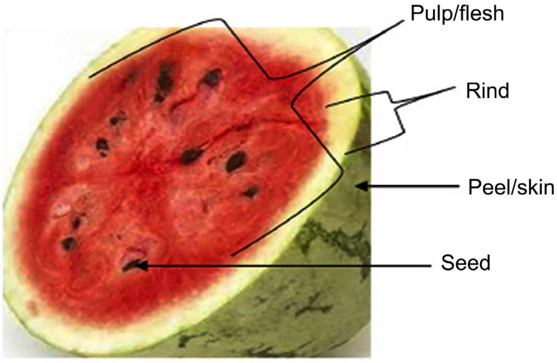

Figure 1. Watermelon biomass.

Biosorption offers the efficiency of low cost and good efficacy too. Thousands of research reports show that materials of biological origin (*i.e.*) bio-sorbents can be used as effective adsorbents to remove different substances such as heavy metals, nutrients, drugs and dyes from aqueous solutions. The use of bio-sorbent has been considered as an alternative to traditional wastewater treatment methods such as chemical precipitation. Bio-sorbent can be used as materials in water treatment systems, adsorbents for chemical or oil spills and other permeable reactive barriers. The precursor material used in this study is the water melon peel/skin also known as rind (see **Figure 1**).

2. Materials/Method

2.1. Sample Preparation/Chemical Activation

Water Melon Rind (WMR) was collected from fruit vendors in Amassoma community, Bayelsa State, Nigeria. It was cut into 4 × 4 cm and washed thoroughly with clean water 12 times, it was then dried at 180˚C in a gas oven for 3 hours and a crispy textured material was obtained. The crunchy biomass was then pulverized into powder using a domestic blender and then the pulverized WMR was passed through a 710 μm sieve and then stored in a well labeled air tight container for further use. Before any treatment the sieved biomass was divided into three portions (A, B, and C) while the last portion is left as it is and labelled as Untreated Watermelon Rind (UWMR).

Portion A: Modification of watermelon rind using distilled water

10 g of untreated biomass was weighed and mixed with 160 mL distilled water in a 250 ml beaker, and the mixture is then subjected to microwave irradiation for 5 minutes. The biomass /distilled water mixture was allowed to cool at room temperature filtered and washed with distilled water. The residue obtained is then dried in an oven for 24 hrs at 60˚C crushed and sieved to particles less than 710 μm. The resulting powder stored in an air tight sample container labelled DWMR.

Portion B: Modification of Watermelon Rind using NaOH solution

10 g of Untreated Watermelon Rind was weighed and mixed with 1M NaOH solution in a 250 mL beaker and the mixture is subjected to microwave radiation for 5 minntes and allowed to cool at room temperature, filtered and washed with distilled water to remove excess reagents (NaOH). The residue obtained is further oven dried at 60˚C for 24 hrs. The dried NaOH treated biomass is then crushed and sieved into particle size less than 710 μm and labeled NaWMR.

Portion C: Modification of watermelon rind using Hydrogen peroxide (H_2O_2)

20 g untreated biomass is weighed into a 250 ml beaker and 170 mL distilled water is added to it. The pH of the mixture is the adjusted to 10 by adding 0.1M NaOH solution. About 65 mL of 30% H_2O_2 is added to the biomass mixture and subjected to Microwave irradiation for 15 mins. The treated biomass is allowed to cool at room temperature and washed with distilled water, and it is later dried in an oven for 24 hours at 60˚C. The final product is crushed and sieved to particles less than 710 μm and labelled as HP-WMR.

2.2. Physicochemical Characterization of Biosorbents

pH measurement

Approximately 1 g of each sample of the bio-sorbent namely, untreated watermelon rind (U-WMR), Watermelon Rind treated with distilled water (D-WMR), Watermelon rind treated with NaOH (Na-WMR) and water melon rind treated with H_2O_2 (HP-WMR) is weighed and put in separate 250 mL beaker that contains 100 mL of distilled water. This was allowed to boil on a hot plate for 5 mins. The solution is later diluted with 200 mL distilled water and allowed to cool. The pH of each sample is then measurement is then using a pH meter and recorded accordingly.

Bulk Density determination

The bulk density of each sample was determined in line with Archimedes' principle. Wherein, a 10 cm^3 measuring cylinder is weighed before (empty) and after it was fully packed with each sample and tapped 2 - 3 times, the difference in weight is determined and noted. Bulk density is calculated by using the following equation:

$$\text{Bulk density} = W_2 - W_1/V \tag{1}$$

where,

W_1 = weight of empty measuring cylinder;

W_2 = weight of the measuring cylinder with sample;

V = volume of cylinder.

Volatile Matter Determination

In order to determine the volatile matter of the bio-sorbents 1 g of each sample is weighed and added to a previously weighed empty crucible. Weights of both the crucible and 1 g of each sample are taken. The set up then placed in an oven for 10 mins at 150˚C after heating, the system is allowed to cool in a desiccator, the volatile matter is calculated using the following expression:

$$\text{Percentage Volatile matter}\left(\text{Vm\%}\right) = \frac{W_{vc}\left(\text{g}\right)}{W_o\left(\text{g}\right)} \times 100 \tag{2}$$

where,

W_{vc} = Weight of the volatile component [weight of empty crucible (W_c)] + weight of sample (1 g);

W_o = Oven dry weight (weight of W_c after oven drying).

Surface Area Determination

The surface area of the bio-sorbents were determined using the Sear's method wherein 0.5 g of each sample was carefully weighed into 250 mL conical flask containing 25 mL HCl (0.1M) (pH 3.50) after which 1 g of NaCl was added to raise the pH to 4, this mixture was then titrated with a standard solution of 0.1M NaOH until a of pH 9 was achieved. The volume needed to increase its pH from 4 to 9 was recorded and used in computing the surface area using Equation (3).

$$\text{Surface area}\left(\text{M}^2/\text{g}\right) = 32V - 25 \tag{3}$$

where, V represents the volume of NaOH used to raise pH from 4 to 9.

Ash Content Determination

The ash content of bio-sorbents was determined using the ASTM D 2866-94 method [15] crucibles were containing the samples were preheated to about 500˚C, and cooled in desiccator, after which it was weighed. 1.0 g of each sample was transferred into the crucible and reweighed. The crucible containing the sample were then placed in the furnace and the temperature was allowed to rise 500˚C for 3 hrs and allowed to cool in desiccator to room temperature and weighed. The ash content was calculated using Equation (4):

$$\text{Ash \%} = \frac{\text{ash weight}}{\text{oven dry weight}} \times 100 \tag{4}$$

Moisture Content Determination

The moisture content of all biosorbent samples were determined using the ASTM D280-33 where by three empty crucibles were first weighed and 1.0 g of each biosorbent sample was added into the crucible. This was done in triplicate. The crucibles containing samples were oven dried at 110˚C to a constant weight for 3 hours. The samples were then placed in a desiccator to cool and re-weight again. The difference between the initial and final mass of the carbon represents the moisture content. The percentage moisture content is determined using the following expression:

$$\text{Moisture \%} = \left(W_1 - W_2 / W_1\right) \times 100 \tag{5}$$

where, W_1 = Initial weight of sample (g);

W_2 = Final weight of sample after drying (g).

3. Results and Discussion

The results of the experimental determination of the physiochemical parameters of the bio-sorbents, Untreated Watermelon Rind (UWMR), Watermelon Rind modified with distilled water (DWMR), Watermelon Rind modified with sodium hydroxide and Watermelon Rind modified with hydrogen peroxide (HP-WMR) are shown in **Figures 2-7**.

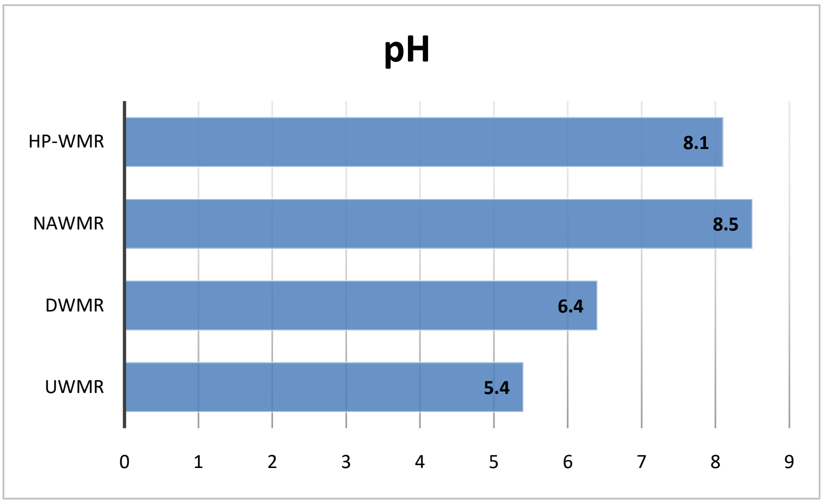

Figure 2. pH values for biosorbents.

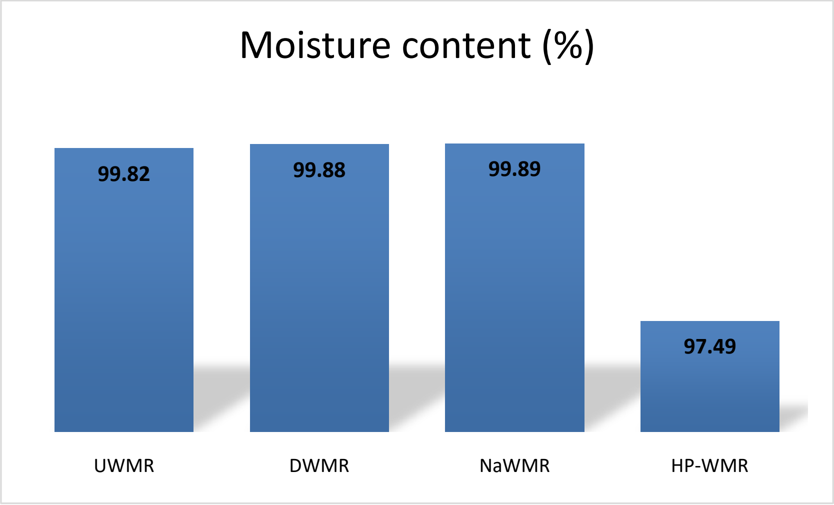

Figure 3. Percentage moisture content of biosorbents.

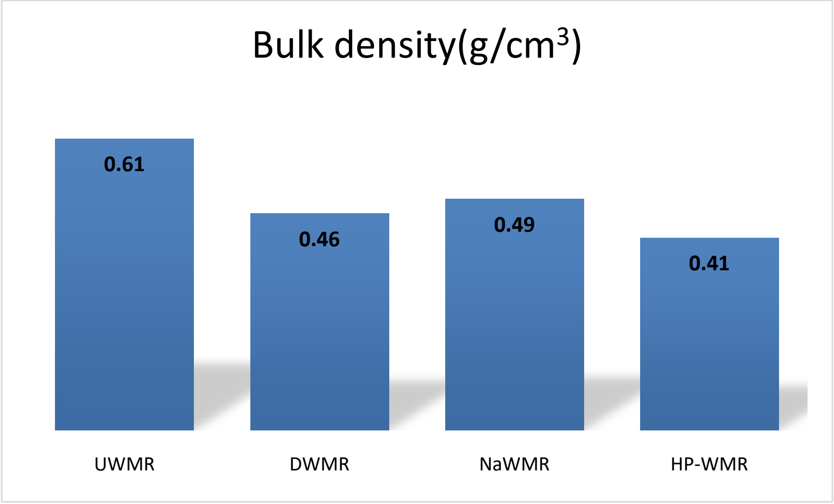

Figure 4. Bulk density of biosorbents.

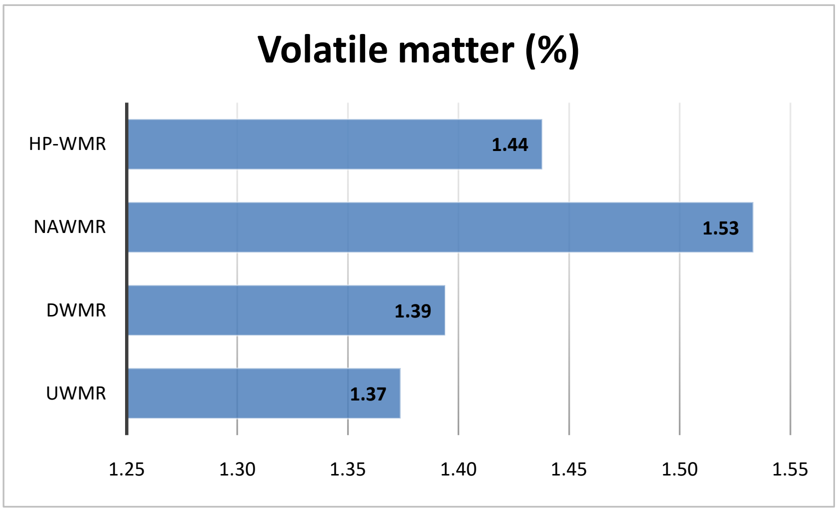

Figure 5. Volatile matter.

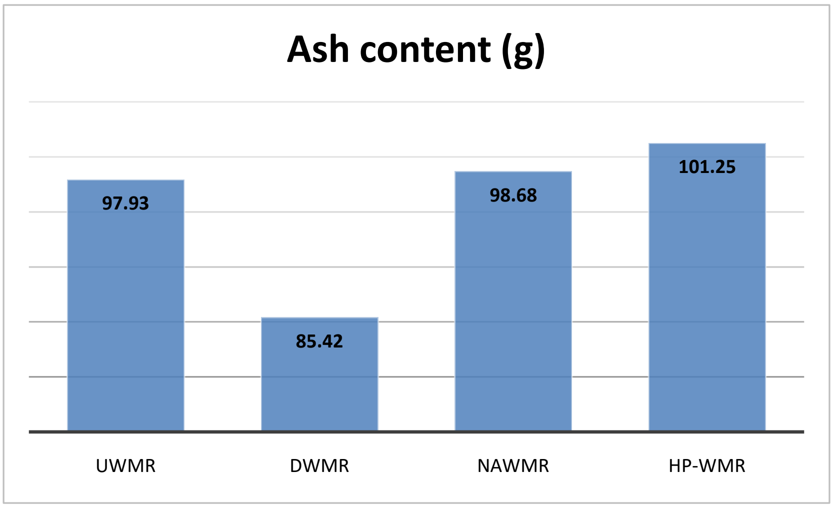

Figure 6. Ash content of biosorbent.

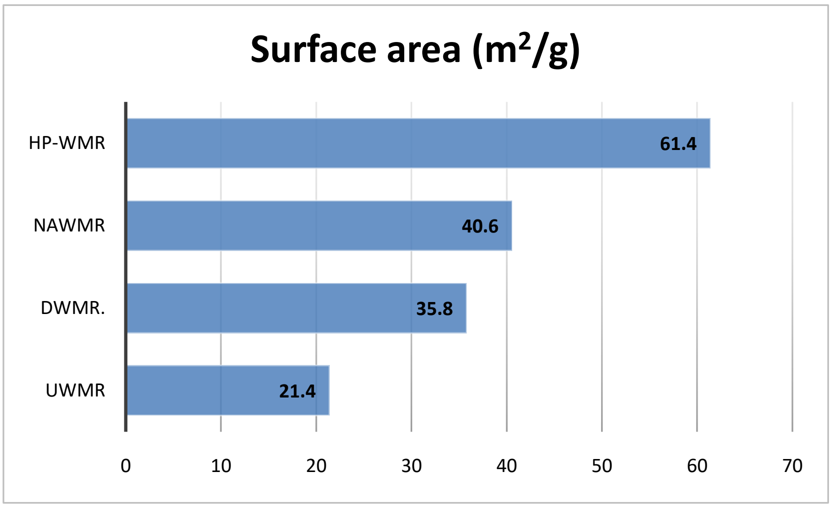

Figure 7. Surface area values for biosorbents.

Scientific investigation has shown that pH influences the charge density around adsorbate and adsorbate molecules [16]. Thus, pH is a crucial parameter that influences the dissociation of active sites on the surface of adsorbents [17].

Figure 2 shows that Untreated Watermelon Rind (UWMR) will be a good adsorbent for anionic species due to its low pH value. Meanwhile, Water Melon Rind treated with distilled water (DWMR) is almost neutral, so it can take both negatively charged and neutral adsorbates. On the other Water Melon Rind treated with sodium hydroxide (NaWMR) is basic, thus its surface is negatively charged, therefore it could serve as a good adsorbent for cationic species such heavy metals. In like manner Water Melon Rind treated with hydrogen peroxide (HP-WMR) has basic pH value which suggests its surface is mostly negatively charged as such it could be a good candidate for the uptake of cationic species such as heavy metals. Research reports have shown that the pH values obtained for NaWMR (8.5) and HP-WMR (8.1) were ideal for adsorption processes as maximum uptake of metals by most activated carbonaceous adsorbents occur between pH 6 - 9 [18].

Moisture is the presence of liquid especially water in trace amount. Scientific reports have shown that moisture content increases linearly with Bulk density [19]. Therefore, high moisture content does not support adsorption while low moisture content enhances adsorption capacity of adsorbents.

From **Figure 3** one can deduce that all four (4) synthesized bio-sorbents (UWMR, DMWR, NaWMR and HP-WMR) have high moisture content (>90%) as such it will be advisable to subject the bio-sorbents to mild heat for some time before they are used as adsorbents, as this will reduce their moisture content and enhance their adsorption capacity [10].

Figure 4 shows the Bulk Density (g/cm^3) obtained for bio-sorbents; UWMR (0.613 g/cm^3), DWMR (0.4563 g/cm^3), NaWMR (0.4867 g/cm^3) and HP-WMR (0.41 g/cm^3). The untreated watermelon rind (UWMR) has the higher bulk density, followed by DWMR, NaWMR and HP-WMR has the least bulk density.

These result shows that all bio-sorbents used in this study have bulk densities that are lower than those found in previous studies [20]. Therefore, bulk densities obtained for the bio-sorbents were within the recommended values for bulk density making them ideal for absorption. However, since bulk density is inversely proportional to surface area, HP-WMR with the least bulk density will perform better than DWMR, NaWMR and UWMR.

From **Figure 5** result from volatile matter shows that NaWMR has the highest value (1.53%) followed by HP-WMR (1.44%), DWMR (1.39%) and the least is UWMR (1.37%).

Ash content is a measure of the total amount of minerals present within a material, whereas the mineral content is a measure of the amount of specific in organic component present within a mineral. Reports have it that higher levels of ash content lessen the general activity of adsorbents because it reduces the effectiveness of the adsorbent in terms of re-use [15]. Results obtained from experiments (see **Figure 6**) suggest the presence of appreciable amount of ash in all the bio-sorbents and this could inhibit surface development [21].

Surface Area

Results from surface area analysis (see **Figure 7**) reveal that UWMR has a surface area of (21.4 m^2/g), DWMR (35.8 m^2/g), NaWMR (40.6 m^2/g) and HP-WMR (61.4 m^2/g). Thus, HP-WMR exhibited the highest surface area as such will have more adsorption sites upon which adsorbate species can be adsorbed. Hence, HP-WMR should be the best adsorbent for adsorption because it has more adsorption site due to its large surface area.

4. Conclusions

Three bio-sorbents were prepared from Watermelon Rind (WMR) using microwave assisted process. The bio-sorbents were then characterized using physiochemical techniques.

Experimental results shows NaWMR (Watermelon Rind modified with Sodium Hydroxide) has a pH of 8.5 and HP-WMR (Watermelon Rind modified with Hydrogen peroxide) has a pH of 8.1, which suggest that NaWMR and HP-WMR possess the appropriate pH values for the uptake of cationic species within aqueous systems.

Experimental results from surface area analysis are UWMR (21.4 m^2/g), DWMR (35.8 m^2/g), NaWMR (40.6 m^2/g) and HP-WMR (61.4 m^2/g). This means that HP-WMR has a larger surface area that can be utilized for adsorption process. Consequently, Bulk density result shows that the unmodified biomass (UWMR) has the highest bulk density and HP-WMR (0.41 g/cm^3) has the lowest bulk density.

Water Melon Rind treated with Sodium hydroxide (NaWMR) has the highest content of volatile matter (1.53%) and HP-WMR (1.3738%) exhibited the least ash content value.

Results from moisture content analysis show that UWMR (99.82%), DWMR (99.88%), NaWMR (99.89%), and HP-WMR (97.49 %) show good similarity in values. Thus, there is a need to expose the bio-sorbents to heat before use as adsorbents. Similarly Results obtained from ash content analysis reveal that NaWMR has the highest (98.63%) followed by UWMR (97.94%), DWMR (85.42%) and HP-WMR (101.25%). The presence of the appreciable amount of ash in all the samples indicates that they can be used as potential sources of organic fertilizer.

The results obtained from this study suggest that chemical modification of Water Melon Rind (WMR) with either distilled water, Sodium Hydroxide or Hydrogen peroxide by means of microwave irradiation, enhances physiochemical properties which could boost the adsorption capacity of Water Melon Rind. Thus, the outcome shows that all the three synthesized bio-sorbents; DWMR, NaWMR and HP-WMR possess the characteristics of a good adsorbent. Accordingly, they can be applied for wastewater treatment process.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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